# NATIONAL BUREAU OF STANDARDS REPORT

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THE NATIONAL BUREAU OF STANDARDS
HIGH TEMPERATURE ABSOLUTE CUT-BAR APPARATUS
FOR DETERMINATION OF THE THERMAL CONDUCTIVITY
OF SMALL SOLIDS

Complementary Report
March 1964

by

D. R. Flynn and H. E. Robinson

to the

Bureau of Ships
Department of the Navy
Washington, D. C.

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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# The National Bureau of Standards High Temperature Absolute Cut-Bar Apparatus for Determination of the Thermal Conductivity of Small Solids

## FORWARD

This report is an unmodified version of a paper given by the authors at the 1963 Thermal Conductivity Conference sponsored by Oak Ridge National Laboratory and held at Gatlinburg, Tennessee, on 16-18 October 1963. It is being made available as an NBS Report in the belief that the contents may be of general interest.

In this report, thermal conductivity data are presented which have not been formally published. All such data are subject to review, editorially and otherwise, prior to formal publication, and their preliminary status should be recognized. The contents of this report may not be referenced.

# THE NATIONAL BUREAU OF STANDARDS HIGH TEMPERATURE ABSOLUTE CUT-BAR APPARATUS

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## ABSTRACT

A description is given of an apparatus used for determining the thermal conductivities of small cylindrical specimens (2.54 cm in diameter and 1.27 cm long) of materials of conductivity in the range 0.01 to 0.1 W/cm deg at temperatures from 100° to 1,200° C. The method utilizes longitudinal heat flow in a small cylindrical sample held between two bars of platinum-rhodium alloy. Measurements have also been made on metal specimens in the form of longer cylinders. Experimental results are presented for 60% platinum--40% rhodium alloy, a nickel-chromium alloy (Inconel 702), a microcrystalline glass (Pyroceram 9606), and a borosilicate glass (Pyrex 7740). A discussion of the test method is given, with attention to possible sources of error.

## INTRODUCTION

We almost wrote an introduction opening with a statement of the object of the work described here and going on to give a justification for undertaking it. In addressing this august body, however, such an introduction would be superfluous.

Of the "methods" papers presented at the previous two conferences, roughly half indicated or implied a definite limitation on specimen size, arising in the main from the inability to procure large samples of adequate homogeneity. In addition to measuring the thermal conductivity using smaller and smaller specimens, most investigators currently are striving for smaller and smaller uncertainties at higher and higher temperatures.

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*Physicist, and Chief, respectively

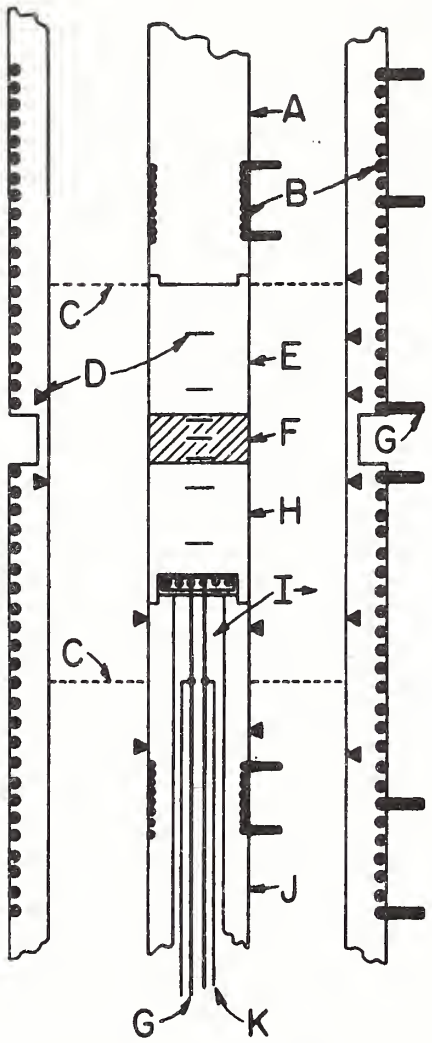


FIGURE 1. SCHEMATIC OF WORKING PARTS OF HIGH TEMPERATURE ABSOLUTE CUT-BAR APPARATUS

A Cold bar support
B Heaters
C Boundaries of region of powder insulation in which heat flow is analyzed
D Thermocouples
E Cold bar
F Specimen
G Current leads to heaters
H Hot bar
I Insulation
J Hot bar support column
K Potential leads to specimen heater

During the initial discussion of the program leading to the apparatus that is being described here, the questions of sample size, temperature range, and limits of uncertainty were discussed at some length. The principal area of discussion involved selection of specimen size, the sponsor wanting us to measure aspirin-tablet-sized specimens while we were inclined toward brick-sized specimens in order to more easily meet the desired accuracy requirements. In compromise, it was decided to develop a method and apparatus for steady-state thermal conductivity measurements at temperatures to 800° C and above, and suitable for solids in the form of small specimens (1/2-in. by 1-in. diameter disks). The overall objective was to provide samples for use by other laboratories as thermal conductivity reference specimens in connection with their measurements on solid semiconductors.

The apparatus being described is a third-generation model, differing from the second-generation model chiefly in that it is built of more refractory materials suitable for temperatures to 1200° C or higher. Because of the shortness of the specimen, an apparatus of the cut-bar type, such as is frequently used in comparative measurements, was selected. To use the contacting bars accurately as heat flow meters, however, entailed numerous complications, not the least of which was lack of a suitable reference standard of appropriate conductivity. There are other problems in the use of contacting bars as meters, some of which have been presented previously at these conferences (1, 2).* For these reasons it was decided that the cut-bar form would be used but the heat flux measurement would be absolute.

## DESCRIPTION OF ABSOLUTE CUT-BAR METHOD

### Apparatus

The working portion of the thermal conductivity apparatus is shown in Figure 1. The 2.54-cm-diameter specimen is interposed between a hot bar and a cold bar, fabricated from 60% platinum--40% rhodium alloy. These bars are supported by an alumina tube from below and an alumina rod from above. Surrounding the inner assembly and concentric with it is an alumina guard cylinder, which is in turn surrounded by a stainless steel case, as shown in Figure 2. The bar assembly is supported on a post fixed to the base of a drill press, in line with the spindle shaft, through which a dead-weight thrust is imposed. The guard and case are supported by the work table of the drill

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*References appear under the heading REFERENCES.

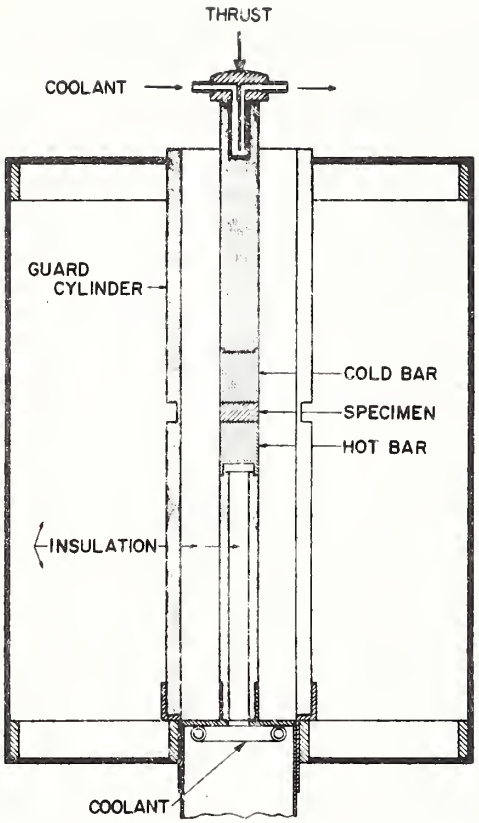


FIGURE 2. SIMPLIFIED SECTION OF THE HIGH TEMPERATURE PORTION OF THE APPARATUS WITH ITS SURROUNDING CASE

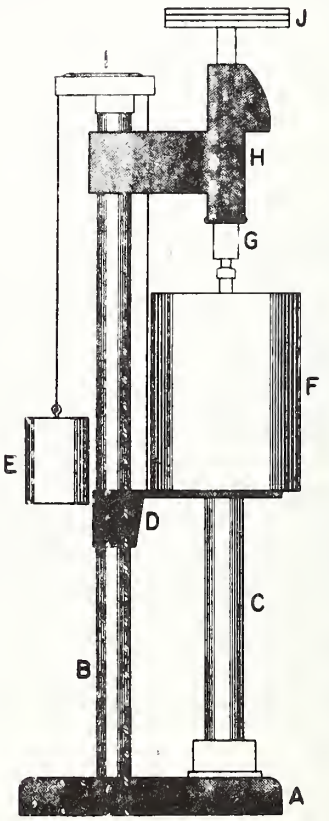


FIGURE 3. OVERALL VIEW OF THE APPARATUS

A Base
B Drill press support column
C Support column for specimen and bar assembly
D Table
E Counterweight
F Guard Case
G Spindle shaft
H Spindle head
I Pulleys
J Dead weights

press, and thus can be lowered to afford access to the bar assembly. Figure 3 is a "patent drawing" view of the overall setup. The space inside the lower alumina support tube, that between the central assembly and the guard cylinder, and that between the guard cylinder and the outer case are filled with finely divided alumina powder as thermal insulation.

The desired temperature distributions within the apparatus are attained with the aid of a number of heaters, each of which was fabricated from 80% platinum--20% rhodium alloy wire.

a. The cold bar is brought to a desired temperature by means of a small heater fabricated from 0.25-mm diameter wire wound in a helical groove around the alumina bar immediately above the cold bar.

b. The hot bar is raised to the desired temperature above the cold bar by means of a small disk-shaped heater (which shall be referred to as the specimen heater) between the hot bar and the lower alumina support tube. This heater was fabricated from 0.25-mm diameter wire and has a resistance of approximately 2 ohms (at room temperature).

c. In order to minimize heat losses down the support tube from the specimen heater, a small heater, fabricated of 0.25-mm wire, was wound on the support tube at a position about 4 cm below the hot bar.

d. A helical groove on the outer surface of the guard cylinder was wound with two separate heaters of 0.5 mm wire. Between the heaters, the guard cylinder wall thickness is reduced to permit a longitudinal temperature distribution along the guard cylinder corresponding to that along the inner assembly. A tap divides each of these heaters into two sections which can be controlled separately.

Temperatures in the system are determined by means of platinum-10% rhodium:platinum thermocouples, which were fabricated from 0.38-mm (unless otherwise stated) reference grade thermocouple wire that had been calibrated by the NBS Temperature Physics Section. Thermocouple positions are shown in Figure 1. There are two thermocouple stations in the hot bar and two in the cold bar. In the hot bar support tube there are two thermocouple stations and one differential thermocouple. Eight thermocouples are installed to define the temperature distribution along the guard cylinder. The number and location of specimen thermocouples are determined by the nature and geometry of the specimen.

Instrumentation

Power for the two guard heaters and the cold bar heater is supplied by variable voltage transformers, which in turn are fed by a voltage regulating transformer. The cold bar heater and the guard heater sections nearest the plane of the specimen are individually regulated by thermocouple-actuated controllers. The guard heater sections remote from the plane of the specimen are manually adjusted.

The specimen heater is powered by a 28-volt, 4-ampere regulated d-c power supply with a small bank of power resistors in series for adjusting to the desired heater current.

The heater in the hot bar support tube is closely regulated, in order to prevent heat gains or losses from the specimen heater along the support tube. Using a small d-c potentiometer, a bias is placed on the signal from a multiple junction differential thermocouple located in the hot bar support tube. The resultant signal is amplified by a breaker-type d-c amplifier and fed into a d-c recorder. The error signal from the control slidewire in the recorder is fed into a current-adjusting-type proportional controller incorporating automatic reset control and rate control. The output of this unit regulates a magnetic amplifier which feeds power to the heater. The operation of a control system of this type has been described in detail by West and Ginnings (3).

All heaters are equipped with separate isolating transformers to minimize current leakage effects.

The noble metal leads of the thermocouples are brought to an isothermal zone box at room temperature. A thermocouple with one junction in the zone box and one in an ice bath is placed in series with a double-pole selector switch, so that each measuring thermocouple is automatically referenced against the ice bath (4). The zone box is also wired to enable determination of the emf developed between similar leads of different thermocouples in the hot bar, in the specimen, and in the cold bar. For these determinations, a separate double-pole selector switch with no ice bath thermocouple in series is used.

Thermocouple emfs are read on a calibrated precision potentiometer, usually to 0.1 μv for thermocouples in the specimen and the hot and cold bar, and to 1.0 μv for thermocouples in the guard cylinder. Power input to the specimen heater is determined by measuring the d-c current through the heater and the voltage drop across potential taps. These measurements are made using calibrated shunt and volt boxes and measuring their output voltages by means of the precision potentiometer.

## ANALYSIS OF METHOD

### Determination of Heat Flow

Ideally, all of the heat generated in the specimen heater should flow through the specimen with no augmentation from other sources. In order to minimize heat flow up or down the hot bar support tube the temperatures at two longitudinal positions about 2.7 cm apart are automatically matched by means of the control system described above. The potential taps used to determine the voltage drop in the specimen heater are located midway between these thermocouple stations as shown in Figure 1. Of the total power output of this heater, only about 0.5 percent is generated in the leads in the region between the potential taps and the uppermost junction of the differential thermocouple; with the two junctions matched in temperature, it is felt that substantially all of the power generated in the leads at positions above the potential taps flows upward toward the specimen. For tests in which the reading of the differential thermocouple is not zero, a correction is made for the slight heat flow through the support column.

Analysis of heat exchanges between the bar assembly and the surrounding powder insulation is essential. Some insight into this problem can be achieved by consideration of a rather crude model. The hot bar, specimen, and cold bar can be thought of as three thermal resistances in series. Shunting each of these resistances is a resistance consisting of a portion of the surrounding powder insulation. For the case in which the thermal conductivity of the specimen is different from that of the bars, there will be abrupt changes in the longitudinal temperature gradients in the bar assembly at the specimen faces. Since the thermal conductivity of the insulation does not change abruptly, the insulation must exchange heat with the bars and specimen to conform to the bar assembly temperatures. In particular, if the specimen has a lower conductivity than the bars, it is necessary for the insulation to steal heat from the bars and specimen on the hotter side and return this heat on the colder side.

It is also necessary to originally establish a matching temperature gradient in the insulation along the bar assembly. In the case where the bars are quite short, this matching gradient may not become established, so that heat exchanges occur along the entire length of the system. Even in the case where the specimen and the bars have the same conductivity, there may be heat interchanges along the entire system. In order to study the parasitic heat flows mentioned, it is necessary to perform a rather extensive analysis.

Although there may be both radial and longitudinal heat flows in the powder insulation, it is the radial temperature gradient at the surface of the bars and specimen that is of particular interest. In the ideal case, the radial gradient at this surface would be zero. This is, however, not the case in general, so that an analysis of the heat flow across this surface must be made. The heat flow across this surface in a longitudinal element of area is

$$dp = 2\pi a k \left(\frac{\partial T}{\partial r}\right)_{r=a} dz \quad , \qquad (1)$$

where a is the radius of the bars and specimen,
k is the thermal conductivity of the powder insulation,
T is the temperature in the powder insulation,
r is the radial coordinate,
z is the longitudinal coordinate.

The net power flowing across the surface r = a between z = 0 and z = $\ell$ is

$$p = 2\pi a \int_0^{\ell} k \left(\frac{\partial T}{\partial r}\right)_{r=a} dz \quad . \qquad (2)$$

In order to evaluate this integral, it is necessary to have a knowledge of the thermal conductivity of the powder insulation and of $(\partial T/\partial r)_{r=a}$ as a function of z. An exact treatment of the system requires solution of the four body composite system involving the two bars, the specimen, and the powder insulation. This has been done by B. A. Peavy of the NBS Heat Transfer Section. The resultant solution is very complicated, however, and was not used for the investigation described in this report.

For this apparatus, $(\partial T/\partial r)_{r=a}$ was determined by analysis of the hollow cylinder of powder insulation with its boundaries at known temperatures. The measured surface temperatures along the bars and the guard cylinders were used to establish functions representing the temperatures on the curved surfaces of the region. The ends of the region, indicated by dashed lines in Figure 1, were assumed to be closed by logarithmic radial temperature distributions, which provide temperature continuity. Axial symmetry was assumed. The analytical solution of the temperature distribution in the region established by these boundary conditions was utilized to evaluate $(\partial T/\partial r)_{r=a}$ in the integral in (2). The thermal conductivity of the powdered alumina insulation was measured in another apparatus (5).

Since the analytical solution of this problem is in the form of an infinite Fourier series, a digital computer is used for numerical evaluation of the data. The details of the mathematical development are not amenable to brief treatment and, hence, are not included in this presentation. A paper (1) giving a fairly detailed analysis of heat flow in the insulation of a simplified cut-bar apparatus was given at an earlier conference.

The total heat flow through the specimen is calculated using the expression

$$Q = P + C\delta + \bar{p} \quad , \qquad (3)$$

where
- Q is the total heat (per unit time) flowing in the specimen
- P is the measured electrical heat input
- C is the heat flow up or down the support column due to a unit temperature difference
- $\delta$ is the temperature difference in the support column as indicated by the differential thermocouple
- $\bar{p}$ is the mean value of the total net heat interchange with the surrounding insulation for a thermocouple span in the specimen (see Equation (2)).

The measured power input, P, is known very accurately. The contribution from heat flow in the support column is minimized by keeping $\delta$ small (usually less than 0.1 deg C). The heaters on the guard cylinder are adjusted to minimize $\bar{p}$.

## Temperature Gradients

Temperatures in the specimens are measured using noble metal thermocouples, as described above. The particular method of thermocouple installation varies somewhat with the specimen being tested, and will be discussed in the appropriate sections describing experimental results.

Since temperature differences in the specimen are rather small, it is essential that the conversion of thermocouple emfs to temperature not introduce any additional uncertainties. The equation

$$E = 16.82614\left(\frac{T}{1000}\right) - 10.30487\left(\frac{T}{1000}\right)^2 + 7.91246\left(\frac{T}{1000}\right)^3 - 2.05305\left(\frac{T}{1000}\right)^4 - 2.85375\left(1.0 - \exp\left[\frac{-4T}{1000}\right]\right) \quad , \qquad (4)$$

where T is temperature (°C) and E is emf (millivolts), was found to fit the platinum--10%rhodium:platinum thermocouple calibration data quite closely over the temperature range 0° to 1450° C. This equation is used for conversion of all thermocouple voltages to temperature.

### Simultaneous Solution of Two Tests

For all tests, thermal conductivity values are computed by simultaneous solution of two tests: 1) an "isothermal" test with no power input to the specimen heater, and 2) a "gradient" test with sufficient power input to the specimen heater to maintain the desired longitudinal temperature gradient. This procedure tends to minimize the effect of certain systematic errors in temperature or heat flow measurements. A brief analysis of the advantages of this simultaneous solution is given in Appendix I. All thermal conductivity values were computed using Equation (A9). The use of this simultaneous solution for computing results significantly improves the precision of the test results and, it is believed, the accuracy as well.

## RESULTS OBTAINED USING THE HIGH TEMPERATURE ABSOLUTE CUT-BAR APPARATUS

In this section, thermal conductivity data are presented which have not been formally published. All such data are subject to review, editorially and otherwise, prior to formal publication.

### Platinum-Rhodium Alloy

Measurements were made of the thermal conductivity of the platinum-rhodium alloy from which the hot and cold bars in the high temperature absolute cut-bar apparatus were to be fabricated.

Description of Specimen. The specimen used for these determinations was in the form of a right circular cylinder 2.539 cm in diameter and 7.5 cm long with recesses at either end. The solid portion of the cylinder was 6.49 cm long. The geometry of this specimen may be envisaged by reference to Figure 1. The specimen was later cut into two pieces which were ground and optically polished to form the hot and cold bars.

The specimen was machined by the manufacturer into the form described. An NBS chemical analysis indicated that the material of the specimen was 60.0 percent platinum and 40.0 percent rhodium by weight*. The specimen was annealed at 1000° C prior to the tests described below.

Test Procedure. The temperature distribution along the platinum-rhodium alloy specimen was determined by means of four 0.38-mm butt-welded platinum-10% rhodium:platinum thermocouples pressed into 0.3-mm grooves in the convex surface of the bar. The distance between the two extreme thermocouples was 3.94 cm.

Thermal conductivity measurements were made in increasing order of temperature from 200° to 1200° C at 200-degree intervals, and then in decreasing order of temperature at 800° and 400° C. Each conductivity determination involved two tests: (1) an "isothermal" test, in which there was no power input to the specimen heater; (2) a "gradient" test with sufficient power input to the specimen heater to maintain a longitudinal temperature gradient in the specimen of about 2 deg/cm. In all of the tests, the guards were adjusted so that there was very little net heat exchange between the specimen bar assembly and the surrounding insulation.

Thermal Conductivity. The thermal conductivity of the platinum-rhodium alloy as determined by simultaneous solution of the various pairs of tests is shown in Figure 4. The points plotted are weighted averages of the values obtained for the three thermocouple spans. Due to uncertainty in the effective distances between thermocouples, there was some scatter in the individual values obtained for the spans. The average of the three values, weighted according to the length of each span, retains only about one-third of this uncertainty.

The circles shown in Figure 4 represent measurements made in increasing order of temperature;** and the inverted triangles those made in decreasing order of temperature. The solid line shown is the quadratic equation of least-mean-squares fit to all of the data points

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*NBS spectrochemical analysis indicated the following impurity elements: Fe 0.08%; Cu, Ir, Pd, Si, Zr 0.001--0.01%; B, Ca less than 0.001%.

**Prior to the tests shown in Figure 4, the specimen was annealed in place at 1000° C; hence the designations "second heating" and "second cooling."

shown. This equation is

$$k = 0.465 + 0.424\left(\frac{T}{1000}\right) - 0.151\left(\frac{T}{1000}\right)^2 , \qquad (5)$$

where k is thermal conductivity expressed in W/cm deg and T is temperature in °C. The dotted lines shown bound the region plus and minus 2.2 percent of the conductivity, equivalent to twice the estimated standard deviation divided by the mean conductivity.

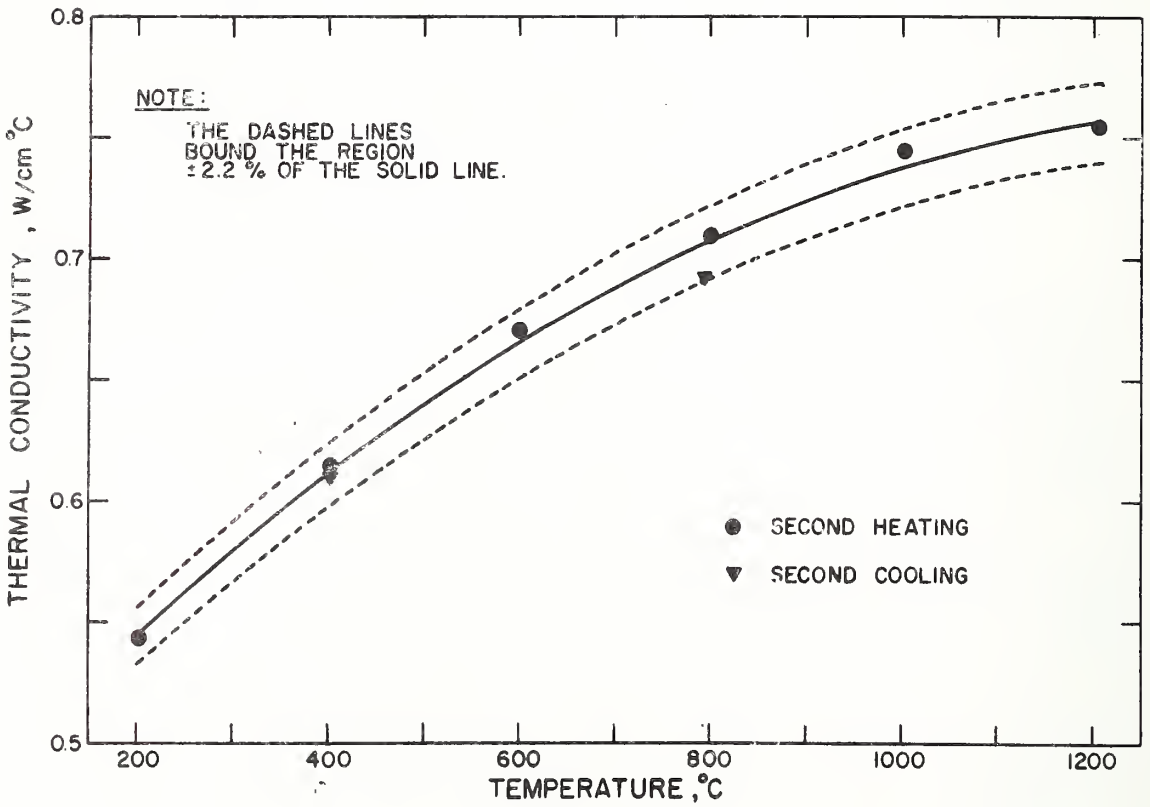


FIGURE 4. THERMAL CONDUCTIVITY OF A 60% PLATINUM--40% RHODIUM ALLOY

Electrical Resistivity and Lorenz Function. When the 60% platinum--40% rhodium alloy bar was purchased, a 0.5-mm wire was drawn by the manufacturer from the same material. The electrical resistivity of a length of this wire was measured in air over the temperature range −195° to 1500° C. Owing mainly to the small cross-sectional area, the values are considered to be uncertain by ±2 percent. The electrical resistivity and thermal conductivity values and the corresponding Lorenz functions are given in Table 1. The apparent Lorenz function appears to be substantially constant near the theoretical value over the temperature range covered.

TABLE 1. THERMAL CONDUCTIVITY, ELECTRICAL RESISTIVITY, AND LORENZ FUNCTION FOR A 60% PLATINUM--40% RHODIUM ALLOY

| t<br>°C | ρ<br>μΩ-cm | k<br>W/cm deg | $L = k\rho/T$<br>$10^8 V^2/deg^2$ | $100(L/L_0-1)$ (a)<br>% |
|---|---|---|---|---|
| −200 | 11.9(b) | --- | --- | --- |
| 0 | 16.8 | --- | --- | --- |
| 200 | 21.6 | 0.544 | 2.48 | +1.5 |
| 400 | 26.4 | 0.611 | 2.40 | −1.8 |
| 600 | 31.4 | 0.665 | 2.39 | −2.1 |
| 800 | 36.5 | 0.708 | 2.41 | −1.5 |
| 1000 | 41.5 | 0.738 | 2.41 | −1.4 |
| 1200 | 46.5 | 0.756 | 2.39 | −2.2 |
| 1400 | 51.6 | --- | --- | --- |
| 1500 | 54.0 | --- | --- | --- |

(a) $L_0 = \frac{\pi^2}{3}\left(\frac{k}{e}\right)^2 = 2.443 \times 10^{-8} V^2/deg^2$, where k = (8.6170 ± 0.0012) x $10^{-5}$ eV/deg is Boltzmann's constant, and e is the electronic charge.

(b) Extrapolated value.

Thermoelectric Power. A thermocouple fabricated from a length of the wire just mentioned and a length of 0.38-mm reference grade platinum wire was calibrated by the NBS Temperature Physics Section over the temperature range 0° to 1100° C. The equation

$$E = 13.25682\left(\frac{T}{1000}\right) - 2.76979\left(\frac{T}{1000}\right)^2 + 6.09233\left(\frac{T}{1000}\right)^3 - 1.79519\left(\frac{T}{1000}\right)^4 - 1.80269\left(1.0 - \exp\left[\frac{-4.5T}{1000}\right]\right), \quad (6)$$

where E is emf (millivolts) and T is temperature (°C), was found to fit the calibration data quite closely.

Differentiation of Equation (6) gives

$$\frac{dE}{dT} = 13.257 - 5.540\left(\frac{T}{1000}\right) + 18.277\left(\frac{T}{1000}\right)^2 - 7.181\left(\frac{T}{1000}\right)^3 - 8.112\exp\left[-\frac{4.5T}{1000}\right] \quad (7)$$

as the thermoelectric power (μV/deg) of 60% platinum--40% rhodium: platinum.

Similarly, differentiation of (4) gives

$$\frac{dE}{dT} = 16.826 - 20.610\left(\frac{T}{1000}\right) + 23.737\left(\frac{T}{1000}\right)^2 - 8.212\left(\frac{T}{1000}\right)^3 - 11.415\exp\left[-\frac{4T}{1000}\right] \quad (8)$$

as the thermoelectric power (μV/deg) of 90% platinum--10% rhodium: platinum.

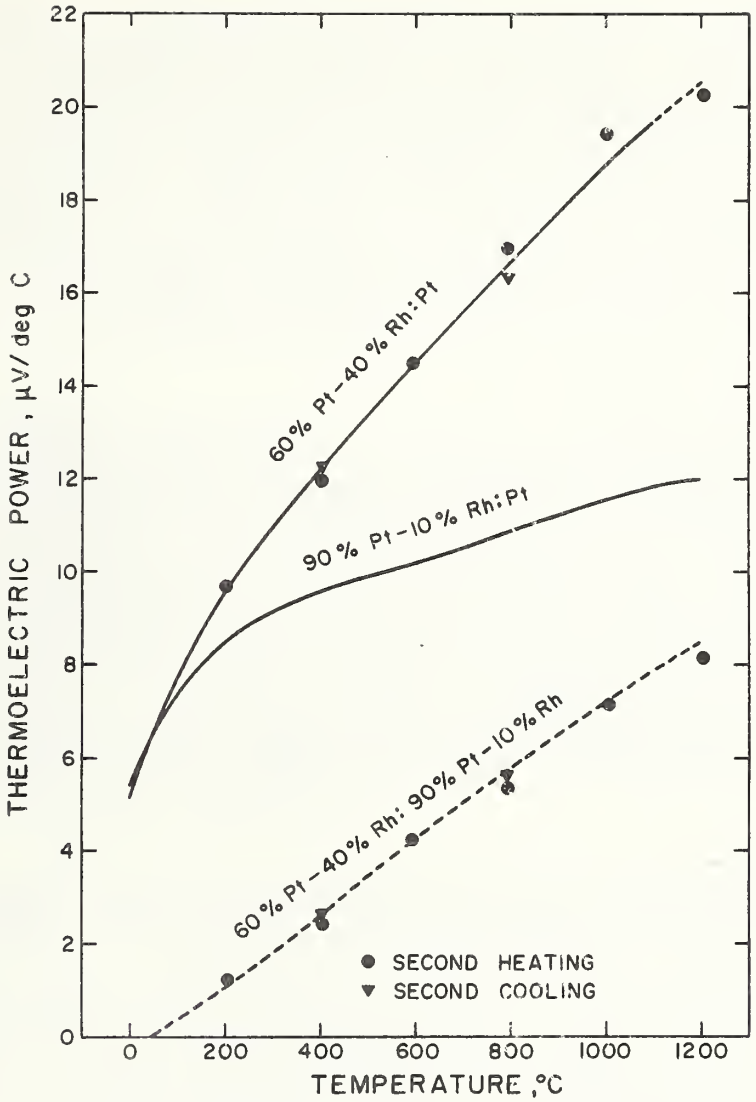


FIGURE 5. THERMOELECTRIC POWER OF PLATINUM--40% RHODIUM AND PLATINUM--10% RHODIUM AGAINST PURE PLATINUM

The solid lines in Figure 5 show the thermoelectric power of 60% platinum--40% rhodium and 90% platinum--10% rhodium against platinum, as computed from (7) and (8), respectively. From the "Law of Intermediate Metals" (6) it follows that the thermoelectric power of 60% platinum--40% rhodium versus 90% platinum--10% rhodium can be obtained by subtracting Equation (8) from (7); the thermoelectric power of this combination is indicated by the dotted line in Figure 5.

For each of the tests presented in Figure 4, for both the upper and lower thermocouple spans of the specimen, emfs were measured between the respective platinum wires of the two thermocouples and between the respective platinum--10% rhodium wires. Thus, the 60% platinum--40% rhodium bar served as the central portion of four differential thermocouples. From the emf outputs of these differential

thermocouples and from the temperature differences, as indicated by the four platinum--10% rhodium:platinum thermocouples, the thermoelectric power of the specimen against 90% platinum--10% rhodium and against platinum was computed. The effects of systematic errors were minimized by simultaneous solution of the "gradient" and "isothermal" tests. In Figure 5, the plotted points give the average values obtained for thermoelectric power as a function of temperature. The circles and triangles correspond to the similar symbols in Figure 4 at the same temperatures. It can be seen that the measured values agree quite well with the curves obtained from equations (7) and (8).

## Nickel-Chromium Alloy

Measurements were made of the thermal conductivity of a nickel-chromium alloy (Inconel 702) which previously had been measured in two models of the NBS metals apparatus (7), in the NBS modified prototype absolute cut-bar apparatus,* and in the NBS steam calorimeter apparatus (8). Determinations made on this specimen material served a dual purpose: (1) since the thermal conductivity of this alloy had previously been measured over a large temperature range, measurements on this material enabled a scrutiny of the accuracy of the high temperature absolute cut-bar apparatus; and (2) they provided additional measurements to elevated temperatures on a metal which was being considered as a possible thermal conductivity reference material.

Description of Specimen. The specimen used for these determinations was in the form of a cylindrical bar nearly identical in size and shape to the platinum-rhodium alloy specimen described above.

The specimen was machined at NBS from the same solution-annealed hot-rolled plate as were the specimens previously measured in several other NBS apparatus. An NBS chemical analysis indicated that the alloy was composed of Ni 79.3%, Cr 17.0%, Al 2.5%, and several other constituents in quantities less than 1 percent (by weight). The detailed analysis is given in Table 2, along with analyses of several similar alloys which will be discussed later.

Test Procedure. Temperatures along the specimen were determined by means of four thermocouples installed in a similar manner to those in the platinum-rhodium alloy.

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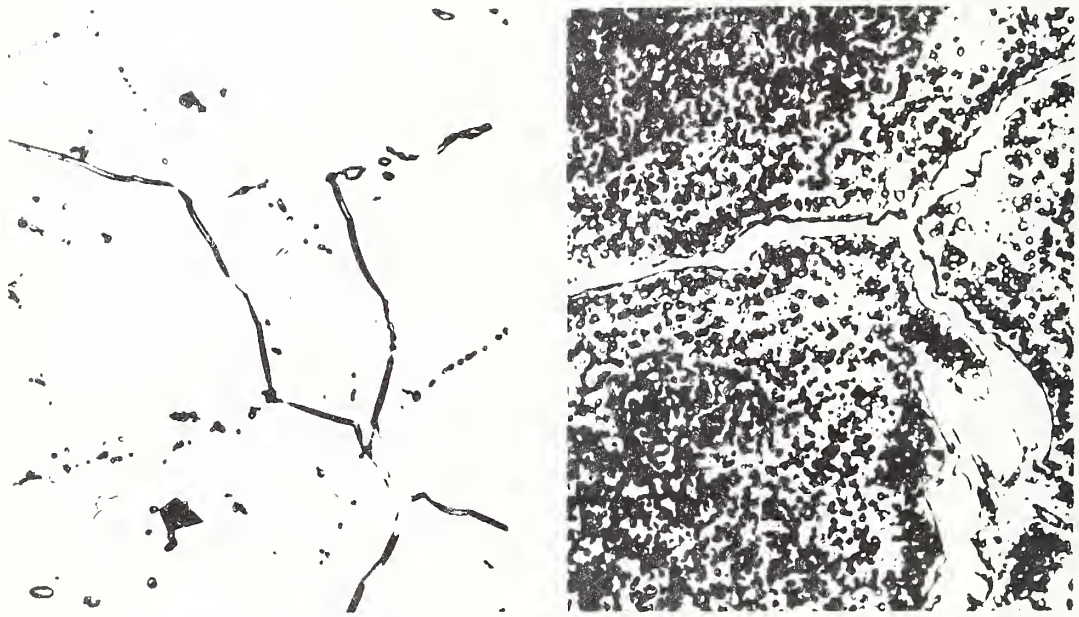
*An earlier model of the present apparatus.

Thermal conductivity measurements were made at 200° C, in increasing order of temperature at 100-degree intervals from 400° to 1200° C, and then in decreasing order of temperature at 900°, 600°, and 300° C. Following preliminary analysis of these data, an additional series of tests was conducted in which measurements were made in increasing order of temperature at 200° C and 300° C, at 50-degree intervals from 400° to 700° C, at 100-degree intervals from 800° to 1200° C, and then in decreasing order of temperature at 900°, 600°, and 300° C. Each conductivity determination involved two tests: (1) an "isothermal" test, and (2) a "gradient" test with sufficient power input to the specimen heater to maintain a longitudinal temperature gradient in the specimen of about 4 or 5 deg/cm.

Thermal Conductivity. The Inconel 702 alloy from which the specimen was fabricated was solution-annealed by the manufacturer prior to purchase by NBS. The reported solution treatment for this alloy is to hold the material at 1975° F (1080° C) for one hour, followed by rapid air cooling. If this alloy is held at temperatures in the range from about 650° to 900° C, age hardening will occur due to precipitation of gamma-prime particles from the super-saturated solid solution. The size of the precipitate is highly dependent on the aging temperature. After 900° C aging, the gamma-prime particles are quite coarse, having a maximum diameter of several thousand Å; if the solution-annealed alloy is aged only at lower temperatures, the precipitate particles are more abundant and finer in size and cannot be resolved by the light microscope.

In the course of the thermal conductivity determinations being discussed, the specimen was cycled twice between room temperature and 1200° C. Since the microstructure of this alloy is dependent on thermal history, the thermal conductivity of the specimen might also be expected to change somewhat, due to heat treatment. At the onset of the measurements, the specimen was in the solution-annealed state, with the gamma-prime phase in supersaturated solution. After the specimen was heated above about 650° C, the gamma-prime phase presumably began to precipitate. As the temperature was further increased, this precipitate became much coarser. Above about 1000° C the precipitate again went into solution. After completion of testing to 1200° C, the specimen was cooled to 900° C, at which time a coarse precipitation occurred. Thus, for the first heating cycle, the specimen was in the solution-annealed state up to about 650° C; from 650° to 900° C, the gamma-prime precipitate was fine at the lower temperatures and became coarser as the temperature was increased; from 1000° to 1200° C, the gamma-prime phase was again in solution. For all of the other tests (first cooling, second heating, second cooling), there



A B

FIGURE 6. MICROSTRUCTURES (X1500) OF INCONEL 702 IN THE SOLUTION-ANNEALED STATE (A) AND IN THE AGE-HARDENED STATE (B)

was a coarse precipitate at 900° C and below. Since the exact temperatures corresponding to precipitation or solution are not known, the temperatures mentioned should be considered approximate. Figure 6 shows the microstructures of this alloy in the solution-annealed, as received, condition and in the age-hardened condition after completion of the described tests.

A cubic equation of least-mean-squares fit was used to represent the thermal conductivity values for the nickel-chromium alloy as determined by simultaneous solution of the various pairs of tests taken during the first heating cycle. This equation is

$$k_s = 0.1198 + 0.07387\left(\frac{T}{1000}\right) + 0.1915\left(\frac{T}{1000}\right)^2 - 0.08265\left(\frac{T}{1000}\right)^3 , \quad (9)$$

where $k_s$ is thermal conductivity (W/cm deg) of the solution-annealed alloy and T is temperature (°C). To enable closer scrutiny of the deviations of individual determinations from the curve, these are shown in Figure 7 as percent departures from the smooth curve plotted against temperature. The dotted lines bound the region plus and minus two standard deviations (estimated) from the solid curve. The maximum departure of any data point from the solid curve is 0.6 percent.

The thermal conductivities obtained when the specimen was in an age-hardened condition can be represented by the equation

$$k_a = 0.1179 + 0.1149\left(\frac{T}{1000}\right) + 0.1173\left(\frac{T}{1000}\right)^2 - 0.05008\left(\frac{T}{1000}\right)^3 , \quad (10)$$

in the same units as above. This equation is that of least-mean-squares fit to the data points obtained during the second heating and second cooling. The deviations of individual determinations are shown in Figure 8 as percent departures from smooth curve (10) plotted against temperature. The dotted lines again bound the region plus or minus two standard deviations (estimated, using data from second heating and cooling tests only).

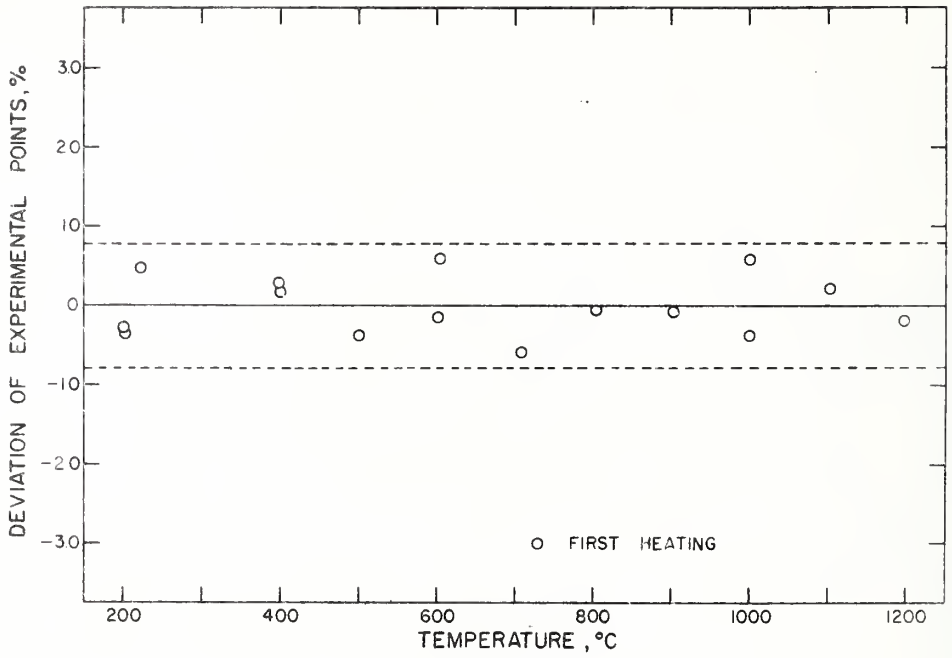



FIGURE 7. DEPARTURES OF THE THERMAL CONDUCTIVITY DATA FOR SOLUTION-ANNEALED INCONEL 702 FROM A SMOOTH CURVE

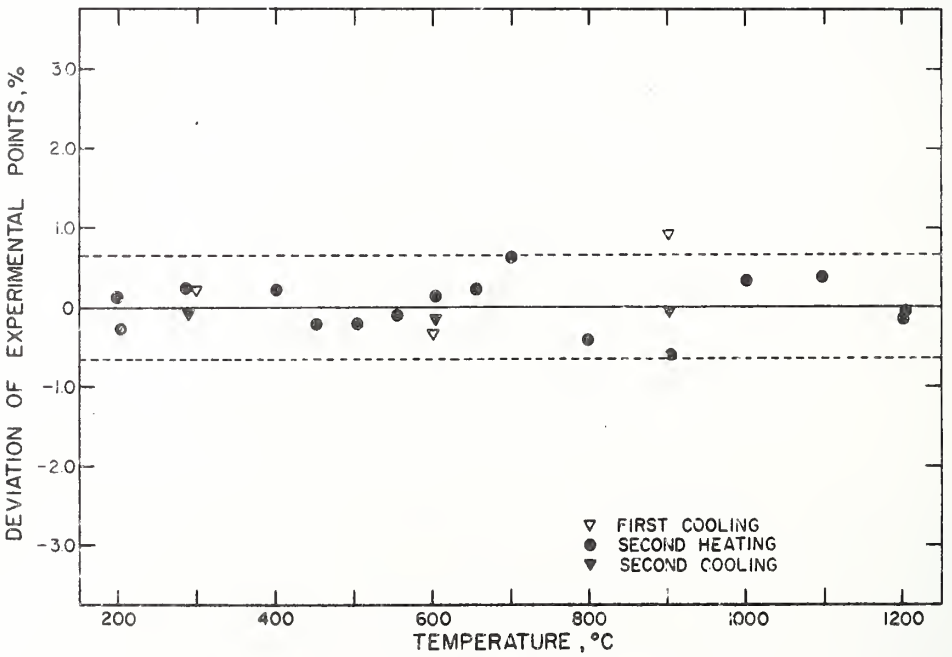



FIGURE 8. DEPARTURES OF THE THERMAL CONDUCTIVITY DATA FOR AGE-HARDENED INCONEL 702 FROM A SMOOTH CURVE

The apparent difference between the thermal conductivity of this alloy in the solution-annealed state and that in the age-hardened state is discussed in another paper being presented to this conference by the same authors, in which some indication is given of an effect on thermal conductivity arising from a cause apparently different from gamma-prime precipitation.

Since all of the results presented in Table 1 are from this laboratory, results obtained on similar alloys by an independent laboratory may also be of interest. Powell and Tye of the National Physical Laboratory recently reported a series of measurements on a group of nickel-chromium alloys somewhat similar in composition to the alloy being investigated at NBS. The chemical composition of the NBS alloy is presented in Table 2 along with the composition of three of the NPL alloys.

TABLE 2. CHEMICAL COMPOSITIONS OF FOUR NICKEL-CHROMIUM ALLOYS, WEIGHT PERCENT

| Constituent | Inconel 702 | Nimonic 75 | Nimonic 80(a) | Nimonic 90 |
|---|---|---|---|---|
| Nickel | 79.3 | 77.9(b) | 73.7(b) | 58.9(b) |
| Chromium | 17.0 | 20.53 | 21.0 | 19.5 |
| Aluminum | 2.5 | --- | 1.2 | 1.4 |
| Titanium | 0.59 | 0.23 | 2.5 | 2.45 |
| Iron | 0.36 | 0.12 | 0.5 | 0.41 |
| Silicon | 0.19 | 0.79 | 0.5 | 0.65 |
| Copper | 0.14 | 0.06 | --- | 0.14 |
| Cobalt | 0.08 | --- | --- | 16.5 |
| Manganese | 0.04 | 0.27 | 0.6 | 0.03 |
| Carbon | 0.066 | 0.126 | 0.04 | 0.06 |

(a) Nominal composition
(b) By difference

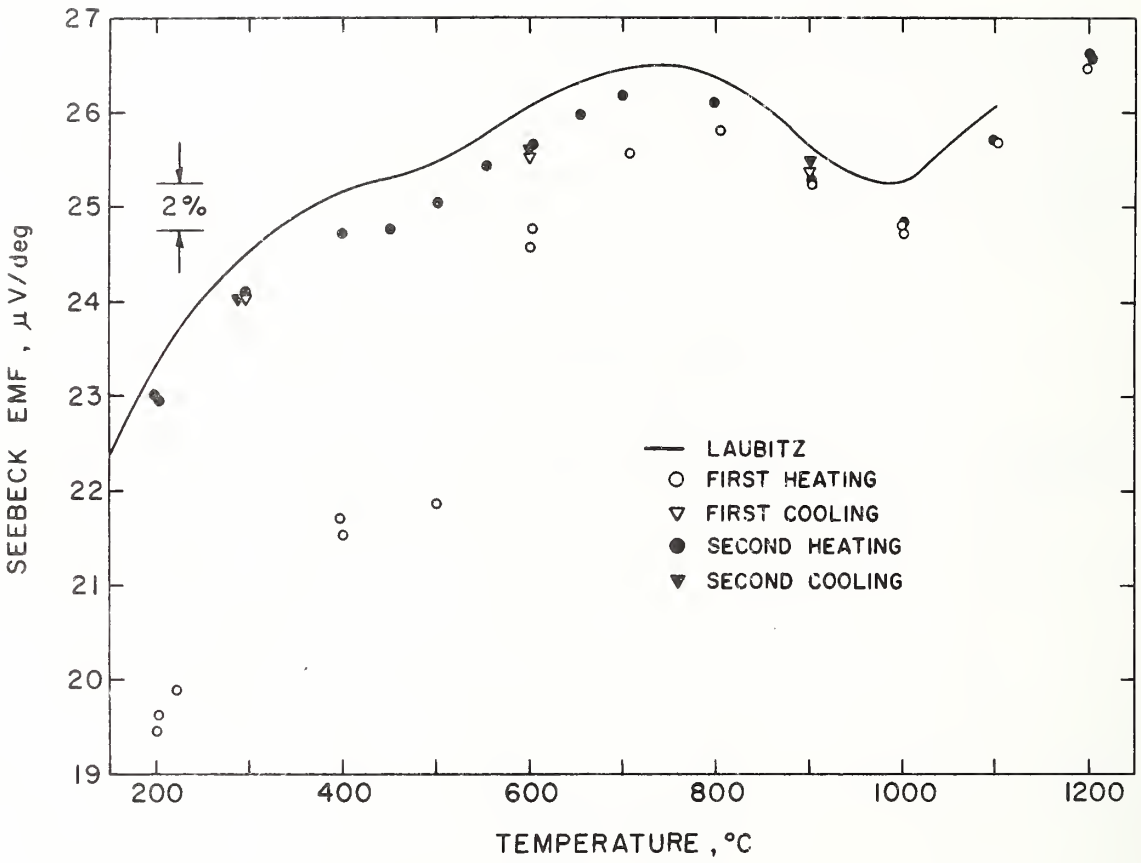



FIGURE 9. THERMOELECTRIC POWER OF INCONEL 702 VERSUS PLATINUM

Table 3 presents tabulated thermal conductivity values for the NBS alloy as determined by this investigation and for the NPL alloys as reported (9). Inspection of Table 3 reveals that the thermal conductivity values for the four alloys have a range, at any given temperature, of about 13 percent at the lower temperatures and that the values converge at higher temperatures so that the range at 800° C is only 3 percent.

TABLE 3. THERMAL CONDUCTIVITY OF FOUR NICKEL-CHROMIUM ALLOYS

| Temp., °C | Inconel 702 | | NPL | NPL | NPL |
|---|---|---|---|---|---|
| | Solution-annealed | Age-hardened | Nimonic 75 | Nimonic 80 | Nimonic 90 |
| 200 | 0.142 | 0.145 | 0.157 | 0.138 | 0.146 |
| 300 | .157 | .162 | .175 | .155 | .165 |
| 400 | .175 | .179 | .191 | .168 | .184 |
| 500 | .194 | .198 | .210 | .184 | .200 |
| 600 | .215 | .218 | .226 | .210 | .218 |
| 700 | .237 | .239 | .243 | .235 | .237 |
| 800 | .259 | .259 | .260 | .255 | .253 |
| 900 | .281 | .280 | ---- | .276 | ---- |
| 1000 | .303 | .300 | ---- | ---- | ---- |
| 1100 | .323 | .320 | ---- | ---- | ---- |
| 1200 | .341 | .338 | ---- | ---- | ---- |

Thermoelectric Power. For each of the tests conducted, emfs were measured between similar legs of the thermocouples in the specimen. Using these data, the thermoelectric power of the specimen against 90% platinum--10% rhodium and against platinum was computed. In Figure 9, the plotted points give the average values for the thermoelectric power of the Inconel 702 specimen against platinum. The symbols correspond to those in Figures (7) and (8). For comparison, we have taken the liberty of also showing the "equilibrium" values for this quantity as presented by M. J. Laubitz to this conference.

Microcrystalline Glass

Measurements were made of the thermal conductivity of a microcrystalline glass (Pyroceram 9606; product of Corning Glass Works, Corning, New York). This material was selected with the hope that it would come close to meeting the following criteria:

1. It should have a thermal conductivity in the range 0.01 to 0.05 W/cm deg, comparable to thermal conductivities of thermoelectric materials.

2. It should be homogeneous and isotropic so that a variety of sizes and shapes of specimens can be made up from one large stock of the material with confidence that all will have the same thermal values.

3. It should be stable up to 1200° C or higher.

4. It should be opaque to thermal radiation up to at least 1200° C.

5. Its cost should not be excessive.

Microcrystalline glass is first formed as a homogeneous glass (incorporating a nucleating agent) which is transparent, so that any defects can be readily detected visually. By suitable heat treatment, the glass is later converted (by Corning) to a polycrystalline solid almost opaque as a result of the large number of very small crystals. The properties of such materials have been described in the literature (10, 11).

Pyroceram 9606 appears to meet the above criteria fairly well, the biggest uncertainty being its stability in the range 800° to 1200° C. There appears to be a small slow change in dimension at the high temperatures, but there is a reasonable possibility that this may not significantly affect the thermal conductivity.

The specimen was fabricated from one of a lot of 2-inch bars supplied by Corning Glass Works to D. C. Ginnings for measurements of thermal diffusivity, the results of which are being presented to this conference by H. W. Flieger. A specimen was also prepared for measurement of thermal conductivity in our metals apparatus (7); the results of these tests are given in another paper being presented to this conference by the present authors.

Description of Specimen. The specimen used for these determinations was in the form of a cylinder 2.540 cm in diameter and 1.269 cm in length. The density of the specimen material was determined at NBS to be $2.599_8$ g/cm$^3$ at 22.71° C. The ends of the specimen were optically polished so as to be flat to within 1/10 light fringe (approx. $3 \times 10^{-6}$ cm).

Test Procedure. The specimen was placed between the platinum-rhodium hot and cold bars, as shown in Figure 1. (The hot and cold bars had been optically polished to be flat to within 1 light fringe.) Temperatures along the Pyroceram specimen were determined by means of three 0.20-mm butt-welded platinum-10% rhodium:platinum thermocouples pressed into 0.15 mm grooves in the convex surface of the specimen. These grooves were equally spaced, one being at the mid-plane of the specimen and the others 0.50 cm above and below the mid-plane.

Thermal conductivity measurements were made in increasing order of temperature from 200° to 500° C at 100-degree intervals. The specimen was then heated to 600° C, but no data were taken (due to a break in the water mains which supplied the cooling water); the specimen was then cooled to room temperature. On second heating, measurements were made at 300° C and at 100-degree intervals from 600° to 1000° C. The specimen was held at 1000° C for about 275 hours, several sets of data being taken during this time. Measurements were then made in decreasing order of temperature at 900°, 600°, and 300° C. Each conductivity determination involved an "isothermal" test and a "gradient" test.

Results of Thermal Conductivity Tests. The smoothed values of thermal conductivity and thermal resistivity of the Pyroceram specimen are shown in our other paper. The values plotted there were calculated from the equation

$$w = 1/k = 26.7 + 9.7\left(\frac{T}{1000}\right) , \qquad (11)$$

where w is thermal resistivity (cm deg/W), k is thermal conductivity (W/cm deg), and T is temperature (°C). Equation (11) is the linear equation of least-mean-squares fit to the thermal resistivity values obtained from the first and second heating cycles. The percent deviations of the experimental resistivity values from the smooth curve are shown in Figure 10. The points connected by a line (or in close juxtaposition) represent the individual values obtained in each pair of

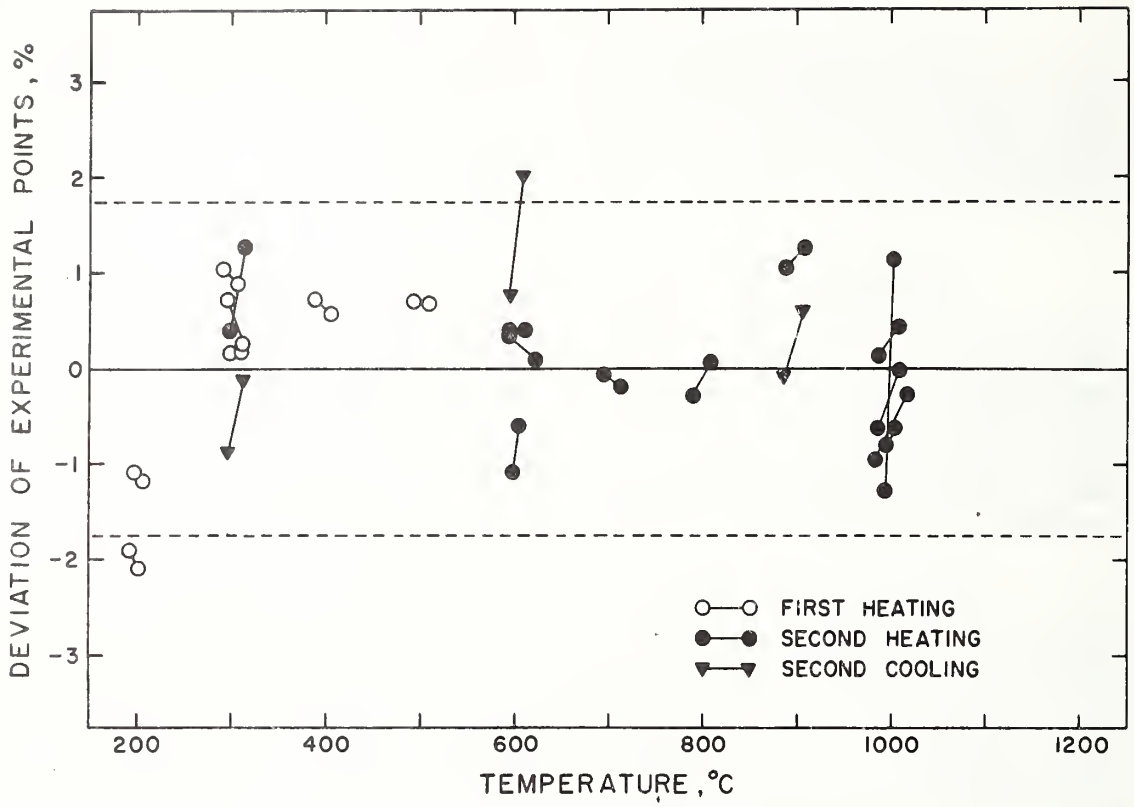



FIGURE 10. DEPARTURES OF THE THERMAL RESISTIVITY DATA FOR PYROCERAM 9606 FROM A STRAIGHT LINE

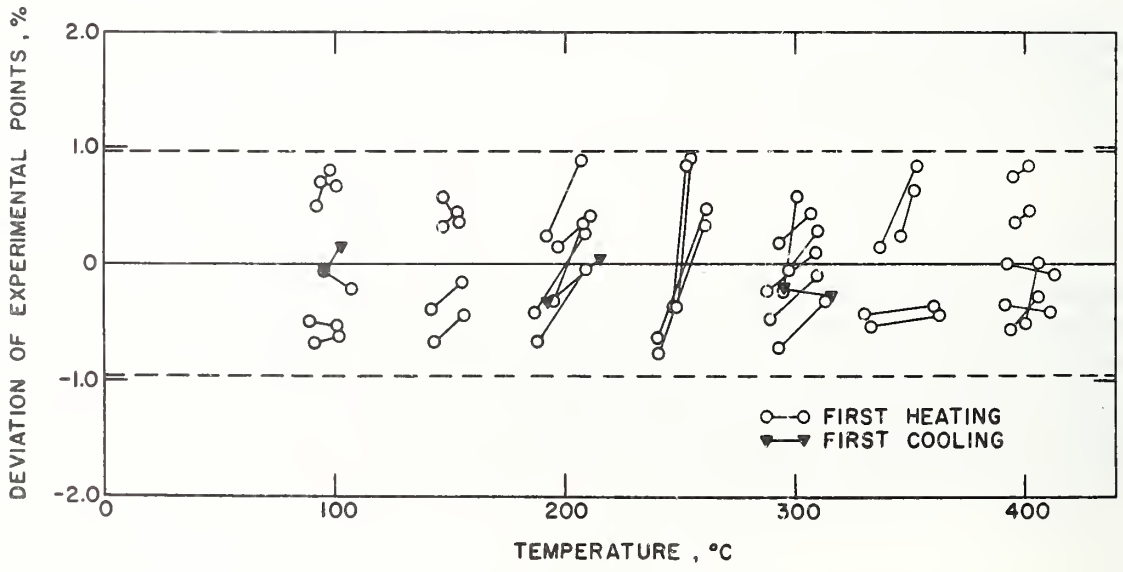



FIGURE 11. DEPARTURES OF THE THERMAL CONDUCTIVITY DATA FOR PYREX 7740 FROM A SMOOTH CURVE

tests for the two thermocouple spans in the specimen. The horizontal displacement of two connected points is indicative of the temperature gradient in the specimen; for most of the tests this gradient was 30 to 45 deg/cm.* The dotted lines in Figure 10 bound the region plus or minus two standard percent deviations (estimated; first and second heating only).

The data points obtained on second cooling are in substantial agreement with the data taken before the specimen was held at 1000° C, thus indicating that no appreciable change in the thermal conductivity of Pyroceram 9606 was perceived as a result of this heat treatment.

## Borosilicate Glass

Measurements were made of the thermal conductivity of a sample of borosilicate glass (Pyrex Code 7740). This material is being investigated as a possible thermal conductivity standard.

Description of Specimen. The specimen used for these determinations was in the form of a right circular cylinder 2.540 cm in diameter and 1.517 cm in length. The specimen was cut from a piece of one-inch commercial plate indicated by the supplier to be from the same original sheet as were specimens prepared for testing in the NBS guarded hot plate (see earlier paper presented to this conference by Robinson and Flynn). The ends of the specimen were optically polished so as to be flat within 1/10 light fringe and then were given a "double-opaque" coating of gold by evaporation technique. The flat surfaces ensured good contact with the hot and cold bars; the gold coating minimized direct radiative transmission through the specimen. After grinding to size but prior to optical polishing, the specimen was carefully annealed by the NBS Glass Section; a 60° prism cut from adjacent material was annealed to the same schedule.** The density of the prism was determined as $2.225_8$ g/cm$^3$ at 24.3° C prior to annealing and as $2.224_6$ g/cm$^3$ at 25.0° C after annealing. The refractive index of this prism was measured on a precision spectrometer at controlled room temperatures

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*Exceptions to this are as follows:

| | |
|---|---|
| First heating: | 200° C, 20 deg C/cm (both pairs); |
| | 300° C, 20 deg C/cm (one pair); |
| Second heating: | 600° C, 60 deg C/cm (one pair); |
| | 600° C, 10 deg C/cm (one pair); |
| | 1000° C, 15 deg C/cm (two pairs); |
| | 1000° C, 70 deg C/cm (one pair). |

**The specimen and prism were heated to 570° C in one hour, held at 570° for two hours, cooled at 1-1/2 deg/hr to 435° C (90 hours), cooled at 10 deg/hr to 355° C (8 hours) and then furnace-cooled.

near 23°C and found to be 1.47257(±0.00003) for the D lines of sodium (5893 Å) prior to annealing; subsequent to annealing the index was found to be 1.47211 (±0.00003).

Test Procedure. The specimen was placed between the platinum--rhodium hot and cold bars, as shown in Figures 1 and 2. Temperatures along the Pyrex specimen were determined by means of three 0.20-mm butt-welded platinum--10% rhodium:platinum thermocouples located in 0.22-mm grooves in the convex surface of the specimen. These grooves were equally spaced, one being at the midplane of the specimen and the others 0.50 cm above and below the mid-plane.

Thermal conductivity measurements were made in increasing order of temperature from 100° to 400° C at 50-degree intervals (Pyrex glass should not be taken above about 450° C unless it can be very, very slowly cooled, since its properties may undergo change). Measurements were then made in decreasing order of temperature at 300°, 200°, and 100° C. Each conductivity determination involved an "isothermal" test and a "gradient" test.

Thermal Conductivity. The thermal conductivities obtained can be represented by the equation

$$k = 11.60 + 16.28\left(\frac{T}{1000}\right) - 32.21\left(\frac{T}{1000}\right)^2 + 51.75\left(\frac{T}{1000}\right)^3 , \qquad (12)$$

where now k is expressed in mW/cm deg and T in °C. The percent departures of the experimental thermal conductivity values are shown in Figure 11. The points connected by a line represent the individual values obtained in each determination for the two thermocouple spans in the specimen; the mid-point of the line represents the mean result of the determination. The dotted lines in Figure 11 bound the region plus or minus two standard deviations (estimated). The points obtained on cooling agree with those taken on heating.

A large number of data points were taken for this specimen in order to study the effects of varying the guarding conditions in the apparatus and the temperature gradient in the specimen. The consistency of the data, as indicated in Figure 11, is excellent, especially when it is considered that for some of these tests the corrections due

to a net heat gain or loss to the insulation were purposely made as high as ten or fifteen percent of the total heat flow. This specimen has not been removed from the apparatus--additional tests are planned. In particular, the effects of using different insulations around the specimen will be studied.

## ESTIMATE OF PRECISION AND ACCURACY

We would like to introduce this section with a quotation from a recent paper (12) by Churchill Eisenhart of the National Bureau of Standards:

> "By the precision of a measurement process we mean the degree of mutual agreement characteristic of independent measurements of a single quantity yielded by repeated applications of the process under specified conditions; and by its accuracy the degree of agreement of such measurements with the true value of the magnitude of the quantity concerned . . . . . accuracy has to do with closeness to the truth; precision, only with closeness together."

We have not yet completed an analysis of all the uncertainties involved in the apparatus being described. The precision of the measurements for a given specimen and installation is indicated by the deviations of the data points from smoothed curves as shown in the results sections above. This component of the precision can probably be improved by further refinement of the corrections and calculation procedure. This is only one aspect of precision, however. Precision also involves the question of how well the results would be reproduced if a new specimen, identical in thermal conductivity, were installed and measurements made. We have not yet done this and hence cannot comment meaningfully as to this aspect of precision, although we feel a reasonable confidence in this regard.

An estimate of accuracy entails evaluation of all systematic errors contained in the measurements. Again quoting Eisenhart (12):

> "The overall systematic error of a measurement process ordinarily consists of elemental systematic errors due to both assignable and unassignable causes. Those of unknown (not thought of, not yet identified, or as yet undiscovered) origin are always to be feared; allowances can be made only for those of recognized origin."

With this in mind, the authors list all of the possible sources of experimental errors which they were able to imagine. In the informal atmosphere of this conference, we invite your comments regarding additional sources of uncertainty and regarding the possible magnitudes of those which are listed.

The "true" value of the thermal conductivity, assuming linear heat flow, is given by

$$k_0 = \frac{(Q - Q')L}{A(\Delta T - \Delta T')} \qquad (13)(A7)$$

where Q and Q' are the total "true" heat flows through the cross-sectional area A in two tests at substantially the same mean temperature, and $\Delta T$ and $\Delta T'$ are the corresponding "true" temperature differences between two parallel planes, normal to the direction of heat flow, a distance L apart (see Appendix I). By independent consideration of the uncertainty in each quantity appearing in Equation (13), an estimate can be made of the resultant uncertainty in thermal conductivity, assuming this equation to be valid. Consideration must also be given to the possibility that heat flow in the specimen may not conform to that assumed in Equation (13); i. e., heat flow in the specimen may not be linear.

Cross-Sectional Area. All thermal conductivity specimens are machined or ground to the form of a right circular cylinder of uniform diameter. The diameter of the specimens is measured with sufficient accuracy that the cross-sectional area at room temperature is uncertain by less than 0.1 percent. No correction for thermal expansion has been applied to the results given in the present paper; the accuracy of such a correction is dependent on the accuracy of the coefficient of linear thermal expansion used and cannot properly be charged against the method of measuring thermal conductivity.

Thermocouple Separation. As discussed previously, all specimen thermocouples are pressed into thin transverse grooves in the convex surface of the specimen. The center-to-center distance between the outermost grooves is determined to within 0.2 percent in the case of metal specimens where this is about 4 cm, and to within 0.5 percent in the case of the short specimens where the separation is about 1 cm. The butt-welded thermocouples are pressed firmly into the grooves and it is felt that the effective separation between two thermocouples very nearly coincides with their center-to-center separation. The maximum possible uncertainty in effective thermocouple separation is one groove

width, i. e., 0.8 percent for a 4 cm span with a 0.03 cm groove and 2.0 percent for a 1 cm span with a 0.02 cm groove; this is considered to be an extremely unlikely occurrence, since it implies that the effective thermocouple positions coincide with the outermost edges of the grooves. A more probable uncertainty level would be intermediate between these extremes. We have arbitrarily doubled the center-to-center uncertainties to allow for possible variations of the effective locations of the thermocouples within the grooves; the estimated probable uncertainties in thermocouple spacing are, then, 0.4 and 1.0 percent for the long and short specimens.

Temperature Difference. There are several sources of uncertainty involved in determining the overall uncertainty in the temperature difference between two positions in the specimen. We invite your attention to the paper (14) given at last year's conference by T. M. Dauphinee for a comprehensive review of the factors involved. As shown in Appendix I and pointed out by Dauphinee, the simultaneous solution of two tests largely eliminates the effects of variations between individual thermocouples. All that is required is that the slope of the temperature versus emf curve be essentially the same for the critical thermocouples. That this is so is indicated by the fact that during an "isothermal test" at 1000°C all specimen thermocouples normally agreed within much better than 0.1%.

It is difficult to estimate the uncertainties involved in converting emf readings into temperatures. Representative thermocouples were given a secondary calibration by the NBS Temperature Physics Section, which with innate conservatism estimates that uncertainties in the calibration are not more than 0.5 degree in the range 0° to 1100° C. However, a plot of departures of the calibration points from the NBS standard tables is smooth within 0.2 degree. The departures of the calibration points from Equation (4), which was used for conversion of all emfs into temperature, are also less than 0.2 degree. We estimate that the uncertainty in calibration is such that conversion of an emf difference to a temperature difference introduces an uncertainty of less than 0.4 percent. Since all thermocouples are pressed firmly into the specimens and are carried around the specimen for some distance in an isothermal region, and since all thermocouples are subjected to essentially identical conditions, the errors due to lead conduction should be negligible and are certainly less than 0.1 percent. For a 10-degree temperature interval, the emf difference was read with less than 0.3 percent uncertainty (for a larger temperature interval this uncertainty is less). Combining these uncertainties according to standard propagation of error formulae (13), the estimated uncertainty in determining a 10-degree temperature difference is 0.5 percent.

Heat Flow Through the Specimen. The volt box, shunt box, and potentiometer used in measuring the electrical power input were each calibrated with an uncertainty of 0.01 percent. A correction is made for current flow through the volt box. Hence the total power input to the heater and leads above the potential taps is measured with an uncertainty of less than 0.05 percent.

About 0.5 percent of the total power is generated in the leads above the potential taps and below the uppermost junction of the differential thermocouple in the hot bar support column. When the two junctions are matched in temperature, substantially all of the heat generated in the leads above the potential taps flows upward toward the specimen and that generated in the leads below the potential taps flows downward. The uncertainty in electrical power input due to heat generation in the current leads is less than 0.3 percent.

As mentioned in the description of the apparatus, there are two absolute thermocouple stations in the hot bar support column and also a differential thermocouple. The former are used in analyzing heat flows to and from the surrounding insulation; the latter is automatically controlled at a null reading in order to prevent heat flow up or down the hot bar support column. It was originally felt that the differential thermocouple would be less subject to errors arising from contamination and hence this couple has been used to evaluate the slight heat flows along the support column. The simultaneous solution procedure used, however, should substantially eliminate the effects of individual variations between the two absolute (i. e., reference temperature at the ice point) thermocouples. In general, the agreement between readings of the temperature difference along the support column using the differential thermocouple and using the two absolute thermocouples is not as good as would be desired. It was thought that the uppermost junctions were sufficiently removed from the specimen heater to be in a substantially isothermal region. In view of the indicated disagreement, however, it appears that there may be angular or radial temperature variations in the support column at the plane of the upper junctions. There is a possibility that the powder insulation in the support column has settled, leaving a cavity in which there is radiation and possibly convection. We intend to investigate this difficulty, both experimentally and analytically. In the meantime it is necessary to assign an additional 1 percent uncertainty due to this unresolved difference.

The problem of heat flow in the insulation surrounding the hot bar--specimen--cold bar assembly was discussed briefly above. In order to evaluate this flow, Equation (2) is used, with the boundary conditions previously discussed. Temperature distributions along the outer surface of the bar assembly and along the inner surface of the coaxial guard cylinder are derived by fitting polynomials through the

measured temperatures along these surfaces. It is believed that these functions serve quite well in defining longitudinal temperature distribution between measured points. The use of logarithmic functions to define the radial temperature distribution across the ends of the hollow cylinder of insulation is believed to be a superior procedure to that of attempting to accurately measure radial temperature variation in a loose-fill insulating material. A mathematical analysis is planned to investigate how sensitive the mathematical model is to the particular end boundary condition assumed.

Temperatures of the guard cylinder are actually measured at locations within the wall of the guard and hence may not correspond exactly to the interior surface temperature. Such radial differences, which result in a slight error in the average temperature of the interior surface temperature of the guard, are essentially the same for two tests at about the same mean temperature and hence cancel out under simultaneous solution. Similarly, angular variations in temperatures around the guard cylinder cancel out if they are the same for both tests. Such is not the case for uncertainties in the longitudinal thermocouple positions, since the temperature distribution along the guard cylinder is different in the two tests. As Figure 2 shows, the bottom of both the hot bar support column and of the guard cylinder are held in a fixed position and the tops are free to move due to thermal expansion. Since the hot bar support column, the rod above the cold bar, and the guard cylinder are all constructed of the same material, they expand together such that relative longitudinal positions are quite well maintained. An investigation of the sensitivity of the apparatus to alignment of components will be made.

In general it is not possible, or at least it is not practical, to manage the temperature distribution along the guard cylinder in such a manner that there is no augmentation or depletion of longitudinal heat flow in the bar assembly. It is necessary, therefore, to have a rather good knowledge of the thermal conductivity of the powder insulation which fills the region between the bar assembly and the guard cylinder. The conductivity of this insulation has been measured separately (5). In addition, it is possible to conduct several tests in which the correction for heat flow through the insulation is varied over a small range about zero. From these data the thermal conductivity of the insulation can be derived using consistency criteria. This has been done in the case of Pyrex glass and significantly improved the precision, and hopefully the accuracy, of the results. In fact, we feel that thermal conductivity values inferred in this manner for the insulation are as good as some values found in the literature resulting from direct measurement.

As can be appreciated, the analysis of heat flow in the insulation is rather complex. Pending further analysis, we will set at 1.5 percent the overall uncertainty introduced into the measurement by

possible departures between the experimental system and the mathematical model, although we feel it is considerably less than this.

Combining all of these uncertainties, the total heat flow through the specimen is believed to be uncertain by less than 2.0 percent. It is believed that this uncertainty can be considerably reduced by further analysis.

Departure From Linear Steady-State Heat Flow. In substantially all acceptable tests, the average temperature variation with time in the specimen does not exceed 0.002 deg/min over the duration of a test, a slow enough variation that heat absorption is less than 0.3 percent of the total power input for the powers used.

Non-linearity of heat flow in the sample would be caused by excessive heat exchanges with the powder insulation in the region of the specimen, or by non-uniformity of contact between the specimen and the hot and cold bars in the case of the short specimens, and by non-uniformity of heat flow near the specimen heater in the case of the long specimens. As far as we know, these effects are negligible but will all be investigated prior to formal publication.

Overall Accuracy. Combining all the known sources of possible uncertainty, according to standard propagation of errors formulae, leads to an overall uncertainty in the thermal conductivity values of less than 2.5 percent for both short and long specimens. This estimate is substantiated somewhat by agreement of results measured in this apparatus with results attained in our laboratory by other methods.

## APPENDIX I

For one-dimensional steady-state heat flow, the total heat flow, Q, through the specimen is

$$Q = -kA\frac{dT}{dz} \quad , \qquad (A1)$$

where k is thermal conductivity,
A is the cross-sectional area of the specimen,
T is the temperature,
z is the longitudinal coordinate.

For moderate temperature ranges, the thermal conductivity of the specimen is assumed to vary linearly with temperature; then (A1) becomes

$$Q = -k_0 A\{1 + \alpha(T - T_0)\}\frac{dT}{dz} \quad , \qquad (A2)$$

where $k_0$ is the thermal conductivity at an arbitrary reference temperature, $T_0$, and $\alpha$ is its corresponding temperature coefficient.

Integration of (A2) yields, assuming Q to be constant,

$$Q = \frac{k_0 A}{L}\Delta T[1 + \alpha(\bar{T} - T_0)] \quad , \qquad (A3)$$

where

$$\Delta T = T_1 - T_2$$
$$\bar{T} = (T_1 + T_2)/2 \quad , \qquad (A4)$$

$T_1$ and $T_2$ being the temperatures at two positions a distance L apart.

Consider two tests at approximately the same mean temperature; let the heat flow in one test be given by (A3) and that in the other test by a similar expression. The difference between these heat flows will be

$$Q - Q' = \frac{k_0 A}{L}[\Delta T - \Delta T' + \alpha\{\Delta T(\bar{T} - T_0) - \Delta T'(\bar{T}' - T_0)\}] \quad , \qquad (A5)$$

where quantities of one test are distinguished from those of the other by use of primes. The expression in braces in (A5) can be set equal to zero by proper choice of the reference temperature, $T_0$.

If

$$T_0 \equiv \frac{\Delta T\bar{T} - \Delta T'\bar{T}'}{\Delta T - \Delta T'} \quad , \qquad \text{(A6)}$$

the thermal conductivity at the indicated reference temperature, $T_0$, is given by

$$k_0 = \frac{(Q - Q')L}{A(\Delta T - \Delta T')} \quad . \qquad \text{(A7)}$$

Equation (A7) gives thermal conductivity in terms of the "true" values of heat flows, temperature differences, and dimensions. Now let

$$\begin{aligned} Q &= q - s & Q' &= q' - s \\ T_1 &= t_1 - \epsilon_1 & T_1' &= t_1' - \epsilon_1 \\ T_2 &= t_2 - \epsilon_2 & T_2' &= t_2' - \epsilon_2 \quad , \end{aligned} \qquad \text{(A8)}$$

where the capital letters designate "true" values of the parameters; the corresponding lower case letters are the measured values for these parameters; and s, $\epsilon_1$, and $\epsilon_2$ are the associated systematic errors. It is assumed that, for two tests at about the same temperature, the systematic errors associated with a particular parameter are the same; experience indicates that this is a good approximation.

From (A7) and (A8) the thermal conductivity is given by

$$k_0 = \frac{(q - q')L}{A(\Delta t - \Delta t')} \quad , \qquad \text{(A9)}$$

corresponding to the mean temperature

$$T_0 = t_0 + \epsilon_0 \quad , \qquad \text{(A10)}$$

where

$$\begin{aligned} t_0 &= \frac{\Delta t\bar{t} - \Delta t'\bar{t}'}{\Delta t - \Delta t'} \\ \epsilon_0 &= \frac{\epsilon_2(t_2 - t_2') - \epsilon_1(t_1 - t_1')}{\Delta t - \Delta t'} \end{aligned} \qquad \text{(A11)}$$

$$\Delta t = t_1 - t_2$$
$$\bar{t} = (t_1 + t_2)/2 \quad . \tag{A12}$$

Equation (A9) gives thermal conductivity in terms of the measured values of the parameters, all systematic errors of the type being discussed having been eliminated. The effective mean temperature will be in error by $\epsilon_0$; however, for most materials the effect of a slight error in mean temperature is negligible.

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